

45900-000717

10/049819  
NEW

INTERNATIONAL APPLICATION NO.	INTERNATIONAL FILING DATE	PRIORITY DATE CLAIMED
PCT/DK00/00455	August 17, 2000	August 19, 1999
TITLE OF INVENTION METHOD AND DEVICE FOR OBJECTIVE QUALITATIVE ANALYSIS OF GRAPE MUSTS AND/OR WINES BY BROADBAND INFRA-RED SPECTROMETRY		
APPLICANT(S) FOR DO/EO/US MARC DUBERNET		

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

- This is a FIRST submission of items concerning a filing under 35 U.S.C. 371.
- This is a SECOND or SUBSEQUENT submission of items concerning a filing under 35 U.S.C. 371.
- This express request to begin national examination procedures (35 U.S.C. 371(f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b) and PCT Articles 22 and 39 (1).
- The US has been elected by the expiration of 19 months from the priority date (Article 31).
- A copy of the International Application as filed (35 U.S.C. 371(c)(2))
  - is transmitted herewith (required only if not transmitted by the International Bureau). WO 00/
  - has been transmitted by the International Bureau.
  - is not required, as the application was filed in the United States Receiving Office (RO/US).
- An English language translation of the International Application as filed (35 U.S.C. 371(c)(2))
  - is transmitted herewith.
  - has been previously submitted under 35 U.S.C. 154(d)(4)
- Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3)).
  - are transmitted herewith (required only if not transmitted by the International Bureau).
  - have been transmitted by the International Bureau.
  - have not been made; however, the time limit for making such amendments has NOT expired.
  - have not been made and will not be made.
- An English language translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).
- An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)).
- An English language translation of the annexes of the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).

Items 11. to 20. below concern document(s) or information included:

- An Information Disclosure Statement under 37 CFR 1.97 and 1.98-1449 and International Search Report (PCT/ISA/210) in German with ( ) references.
- An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
- A FIRST preliminary amendment.
- A SECOND or SUBSEQUENT preliminary amendment.
- A substitute specification.
- A change of power of attorney and/or address letter.
- A computer-readable form of the sequence listing in accordance with PCT Rule 13ter.2 and 35 U.S.C. 1.821-1.825.
- A second copy of the published international application under 35 U.S.C. 154(d)(4).
- A second copy of the English language translation of the international application under 35 U.S.C. 154(d)(4).
- Other items or information:
  - Two (2) sheets of Formal Drawings
  - 2

U.S. APPLICATION NO (if known, see 37 CFR 1.5) NEW 10/049819		INTERNATIONAL APPLICATION NO PCT/DK00/00455	ATTORNEY'S DOCKET NUMBER 45900-000717																
		CALCULATIONS PTO USE ONLY																	
<p>21. <input checked="" type="checkbox"/> The following fees are submitted:</p> <p><b>BASIC NATIONAL FEE (37 CFR 1.492(a)(1)-(5):</b> Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO and International Search Report not prepared by the EPO or JPO. .... <b>\$1,040.00</b></p> <p>International preliminary examination fee (37 CFR 1.482) not paid to USPTO but International Search Report prepared by the EPO or JPO. .... <b>\$890.00</b></p> <p>International preliminary examination fee (37 CFR 1.482) not paid to USPTO but international search fee (37 CFR 1.445(a)(2)) paid to USPTO. .... <b>\$710.00</b></p> <p>International preliminary examination fee (37 CFR 1.482) paid to USPTO but all claims did not satisfy provisions of PCT Article 33(1)-(4). .... <b>\$690.00</b></p> <p>International preliminary examination fee (37 CFR 1.482) paid to USPTO and all claims satisfied provisions of PCT Article 33(1)-(4). .... <b>\$100.00</b></p>																			
<b>ENTER APPROPRIATE BASIC FEE AMOUNT =</b>																			
<p>Surcharge of <b>\$130.00</b> for furnishing the oath or declaration later than <input type="checkbox"/> 20 <input checked="" type="checkbox"/> 30 months from the earliest claimed priority date (37 CFR 1.492(e)).</p> <table border="1" style="width: 100%; border-collapse: collapse;"> <thead> <tr> <th>CLAIMS</th> <th>NUMBER FILED</th> <th>NUMBER EXTRA</th> <th>RATE</th> </tr> </thead> <tbody> <tr> <td>Total Claims</td> <td>23 - 20 =</td> <td>3</td> <td>X \$18.00</td> </tr> <tr> <td>Independent Claims</td> <td>2 - 3 =</td> <td>0</td> <td>X \$80.00</td> </tr> <tr> <td colspan="2">MULTIPLE DEPENDENT CLAIM(S) (if applicable)</td> <td>None</td> <td>+ \$270.00</td> </tr> </tbody> </table>				CLAIMS	NUMBER FILED	NUMBER EXTRA	RATE	Total Claims	23 - 20 =	3	X \$18.00	Independent Claims	2 - 3 =	0	X \$80.00	MULTIPLE DEPENDENT CLAIM(S) (if applicable)		None	+ \$270.00
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<b>TOTAL OF ABOVE CALCULATIONS =</b> <b>\$ 1074.00</b>																			
<p><input type="checkbox"/> Applicant claims small entity status. See 37 CFR 1.27. The fees indicated above are reduced by 1/2.</p> <table border="1" style="width: 100%; border-collapse: collapse;"> <thead> <tr> <th colspan="2"></th> <th style="text-align: center;"><b>SUBTOTAL =</b></th> <th style="text-align: center;"><b>\$ 1074.00</b></th> </tr> </thead> <tbody> <tr> <td colspan="2">Processing fee of <b>\$130.00</b> for furnishing the English translation later than <input type="checkbox"/> 20 <input type="checkbox"/> 30 months from the earliest claimed priority date (37 CFR 1.492(f)).</td> <td style="text-align: center;">+</td> <td style="text-align: center;"><b>\$ 0</b></td> </tr> </tbody> </table>						<b>SUBTOTAL =</b>	<b>\$ 1074.00</b>	Processing fee of <b>\$130.00</b> for furnishing the English translation later than <input type="checkbox"/> 20 <input type="checkbox"/> 30 months from the earliest claimed priority date (37 CFR 1.492(f)).		+	<b>\$ 0</b>								
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<p>Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). <b>\$40.00</b> per property</p> <table border="1" style="width: 100%; border-collapse: collapse;"> <thead> <tr> <th colspan="2"></th> <th style="text-align: center;"><b>TOTAL FEES ENCLOSED =</b></th> <th style="text-align: center;"><b>\$ 1074.00</b></th> </tr> </thead> <tbody> <tr> <td colspan="2"></td> <td style="text-align: center;">+</td> <td style="text-align: center;">Amount to be: refunded <b>\$</b> charged <b>\$</b></td> </tr> </tbody> </table>						<b>TOTAL FEES ENCLOSED =</b>	<b>\$ 1074.00</b>			+	Amount to be: refunded <b>\$</b> charged <b>\$</b>								
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<p>a. <input type="checkbox"/> A check in the amount of <b>\$</b> _____ to cover the above fees is enclosed.</p> <p>b. <input checked="" type="checkbox"/> Please charge my Deposit Account. No. 08-0750 in the amount of <b>\$1074.00</b> to cover the above fees. A triplicate copy of this sheet is enclosed.</p> <p>c. <input checked="" type="checkbox"/> The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. <u>08-0750</u>.</p>																			
<p><b>NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the application to pending status.</b></p>																			
<p>Send all correspondence to <b>Harness, Dickey &amp; Pierce, P.L.C - Customer No. 30593</b> <b>Post Office Box 8910</b> <b>Reston, Virginia 20195</b></p>																			
<p>Date: <u>02/19/02</u></p>																			
<p>By <u>Donald J. Daley</u> Donald J. Daley</p>																			
<p>DJD/kna Revised 2/1/02</p>																			

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Application No.: To be Assigned

Filing Date: February 19, 2002

Applicant: Marc Dubernet

Group Art Unit: Unknown

Examiner: Unknown

Title: METHOD AND DEVICE FOR OBJECTIVE QUALITATIVE  
ANALYSIS OF GRAPE MUSTS AND/OR  
WINES BY BROADBAND INFRA-RED  
SPECTROMETRY

Attorney Docket: 45900-000717

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Box Amendment  
Commissioner of Patents and Trademarks  
Washington, D.C. 20231

**PRELIMINARY AMENDMENT**

Sir:

The following Preliminary Amendments and Remarks are respectfully submitted  
in connection with the above-identified application.

This application is the National Phase under 35 USC §371 of PCT International  
Application No. PCT/DK00/00455 which has an International filing date of August 17, 2000,  
which designated the United States of America the entire contents of which are hereby  
incorporated by reference.

**IN THE CLAIMS**

Please cancel all of claims 1-23 pending in the International Application.

Please add the following new claims:


Please replace International Applications claims with new claims 24-46 attached hereto, for prosecution in connection with the present U.S. National Stage application.

**REMARKS**

Per the present Preliminary Amendment, all of the pending International claims have been canceled. In substitution therefore, Applicant submits new claims 24-46 attached hereto, for prosecution in connection with the present U.S. National Stage application. An early indication of the allowability of each of these new claims 24-46 in connection with the present application earnestly solicited.

Should there be any outstanding matters that need to be resolved in the present application, the Examiner is respectfully requested to contact Donald J. Daley at the telephone number of the undersigned below.

If necessary, the Commissioner is hereby authorized in this, concurrent, and future replies, to charge payment or credit any overpayment to Deposit Account No. 08-0750 for any additional fees required under 37 C.F.R. § 1.16 or under 37 C.F.R. § 1.17; particularly, extension of time fees.

Respectfully submitted,

Dated: 2/19/02

By: Donald J. Daley  
Donald J. Daley  
Reg. No. 34,313

HARNESS, DICKEY & PIERCE, P.L.C.  
P.O. Box 8910  
Reston, Virginia 20195  
(703) 390-3020

## PATENT CLAIMS

24. A method for the objective qualitative analysis of liquid vinification compositions, *characterised in that:*

- an initial configuration and calibration step (13) is carried out during which:  
5 - a group of characteristic parameters are selected, the characteristic parameters being capable of characterising the quality of a vinification composition to be analysed, and comprising the concentration levels in the vinification composition of characteristic compounds, chosen among at least a compound produced by *Botrytis cinerea*, at least a compound produced by *yeasts*, and at least a compound produced by acetic bacteria, and at least a compound produced by lactic acid bacteria,

10 - calibration values ( $\alpha_i$ ,  $K_i$ ,  $B_i$ ) of spectroscopic criteria are determined and recorded for each characteristic parameter, the spectroscopic criteria being chosen to enable evaluation of the characteristic parameter in a vinification composition from an infrared absorption spectrum, which can be generated from a sample of this vinification composition,

- for each vinification composition to be analysed:  
15 - a spectroscopic analysis (17, 18) is carried out during which a continuous infrared absorption spectrum of a sample of this vinification composition is generated,  
- the spectroscopic criteria of each characteristic parameter are applied to the said continuous spectrum in such a way that the value ( $V_i$ ) of this characteristic parameter in the vinification composition is evaluated by automatic calculation (19).

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25. A method according to claim 24, *characterised in that* the group of characteristic parameters comprises the concentration of each of the characteristic compounds, belonging to the group consisting of gluconic acid, ethanal, ethyl acetate, arabitol, mannitol, sorbitol, 2,3-butanediol, methyl-3 butanol-1, glycerol, mesoinositol and isoamyl acetate.

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26. A method according to claim 24, *characterised in that* the continuous spectrum is generated by Fourier transform interferometric spectroscopy.

27. A method according to claim 24, *characterised in that* the spectroscopic criteria are chosen to enable evaluation of each characteristic parameter in a vinification composition from a near and mid infrared absorption spectrum, and in that the near and mid infrared continuous spectrum of each vinification composition to be analysed is generated.

**28.** A method according to claim 24, *characterised in that* at least an objective quality index ( $Q_i$ ) of the vinification composition is calculated as a function of the said value ( $V_p$ ) evaluated from at least a characteristic parameter of the vinification composition to be analysed.

5       **29.** A method according to claim 28, *characterised in that* the function is a polynomial function.

**30.** A method according to claim 28, *characterised in that* there are calculated:

- a first quality index, representing the attack on the harvest by *Botrytis cinerea*, at least as a function of the value of concentration levels of gluconic acid, mannitol and sorbitol evaluated from the 10 continuous spectrum by application of spectroscopic criteria,

- a second quality index, representing the attack by yeasts, at least as a function of the value of the concentration levels of ethanal, ethyl acetate, arabinol, of 2,3-butanediol, of methyl-3 butanol-1, of glycerol, of mesoinositol and of isoamyl acetate evaluated from the continuous spectrum by application of spectroscopic criteria,

15       - a third quality index representing the attack by acetic bacteria, at least as a function of values of the concentration levels of acetic acid, ethyl acetate and of 2,3-butanediol evaluated from the continuous spectrum obtained by application of spectroscopic criteria,

- a fourth quality index representing the attack by lactic acid bacteria, at least as function of the value of concentration levels of lactic acid, mannitol and of 2,3-butanediol, evaluated from the 20 continuous spectrum obtained by application of spectroscopic criteria,

- and a fifth quality index representing the fermentation ability, at least as a function of the value of the concentration of mesoinositol.

**31.** A method according to claim 28, *characterised in that* each characteristic parameter is 25 evaluated and each quality index ( $Q_i$ ) is calculated by data processing immediately after the spectroscopic analysis step has been carried out.

**32.** A method according to claim 24, *characterised in that* the spectroscopic criteria comprise for each characteristic parameter, for a selection of an integral number  $N$  of spectral bands  $\alpha_i$  of 30 predetermined wavelengths, the value of the luminescence spectral density  $Li$  of the said continuous spectrum, and in that a value  $V_p$  of the characteristic parameter is calculated according to the formula:

$$V_p = Bo + \sum_{i=1}^N Ki Li$$

where  $Bo$  and  $Ki$  are predetermined coefficients.

33. A method according to claim 32, characterised in that each N lies between 5 and 30.

34. A method according to claim 34, characterised in that the group of characteristic parameters further comprises alcoholic volumetric strength; total sugar content; total acidity; pH; concentration of 5 acetic acid; concentration of malic acid; concentration of tartaric acid; concentration of lactic acid; and content of phenolic compounds.

35. A device for objective qualitative analysis of liquid vinification compositions, characterised in that it comprises :

10 memory means (8) in which the calibration values (( $\sigma_i$ ,  $K_i$ ,  $B_i$ )) of spectroscopic criteria are recorded for a group of characteristic parameters capable of characterising the quality of a vinification composition to be analysed, this group comprising the concentration levels in the vinification composition of characteristic compounds selected from at least a compound produced by *Botrytis cinerea*, at least a compound produced by yeasts, and at least a compound produced by acetic bacteria, 15 at least a compound produced by lactic acid bacteria, the said spectroscopic criteria being selected for each characteristic parameter to enable evaluation of the characteristic parameter in a vinification composition from an infrared absorption spectrum that can be generated from a sample of this vinification composition,

20 spectroscopic analysis means (1, 2, 3, 4, 6) for collecting a sample of vinification composition and generating an infrared continuous spectrum of this sample,

calculation means (7) arranged to apply the spectroscopic criteria of each characteristic parameter to the said continuous spectrum and to evaluate by automatic calculation the value ( $V_p$ ) of 25 this characteristic parameter in the vinification composition.

36. A device according to claim 35, characterised in that the group of characteristic parameters comprises the concentration of each of the characteristic compounds belonging to the group consisting of gluconic acid, ethanol, ethyl acetate, arabinol, mannitol, sorbitol, 2,3-butanediol, methyl-3 30 butanol-1, glycerol, mesoinositol and isoamyl acetate.

37. A device according to claim 35, characterised in that the spectroscopic analysis means (1, 2, 3, 4, 6) comprise a Fourier transform interferometric spectrometer.

35 38. A device according to claim 35, characterised in that the selection criteria are chosen for each characteristic parameter to enable evaluation of the characteristic parameter in a vinification composition from a near and mid infrared spectrum,

and in that the spectroscopic analysis means (1, 2, 3, 4, 6) are arranged to generate near and mid infrared continuous absorption spectra.

39. A device according to claim 35, characterised in that the calculation means (7) are arranged  
5 to calculate automatically at least an objective quality index ( $Q_j$ ) of the vinification composition as a function of this value ( $V_p$ ) evaluated from at least a characteristic parameter for the vinification composition to be analysed.

40. A device according to claim 39, characterised in that the function is a polynomial function.  
10

41. A device according to claim 39, characterised in that the calculation means (7) are arranged to calculate:  
- a first quality index, representing the attack on the harvest by *Botrytis cinerea*, at least as a function of the value of concentration levels of gluconic acid, mannitol and sorbitol evaluated from the continuous  
15 spectrum by application of spectroscopic criteria,  
- a second quality index, representing the attack by yeasts, at least as a function of the value of the concentration levels of ethanol, ethyl acetate, arabitol, of 2,3-butanediol, of methyl-3 butanol-1, of glycerol, of mesoinositol and of isoamyl acetate evaluated from the continuous spectrum by application of spectroscopic criteria,  
20 - a third quality index representing the attack by acetic bacteria, at least as a function of values of the concentration levels of acetic acid, ethyl acetate and of 2,3-butanediol evaluated from the continuous spectrum obtained by application of spectroscopic criteria,  
- a fourth quality index representing the attack by lactic acid bacteria, at least as function of the value of concentration levels of lactic acid, mannitol and of 2,3-butanediol, evaluated from the continuous  
25 spectrum obtained by application of spectroscopic criteria,  
- and a fifth quality index representing the fermentation ability, is calculated at least as a function of the value of the concentration of mesoinositol.

42. A device according to claim 39, characterised in that the calculation means (7) are arranged  
30 to evaluate each characteristic parameter and to calculate each quality index  $Q_j$  by data processing immediately after the generation of the continuous spectrum by the spectroscopic analysis means (1, 2, 3, 4, 6), and to provide the results of these calculations to means (10) that are read by a user.

43. A device according to claim 42, characterised in that the means (10) that are read comprise  
35 means for printing a report of results.

44. A device according to claim 35, *characterised in* that for each characteristic parameter, the calibration values comprise an integral number N of spectral bands  $\sigma_i$  of predetermined wavelengths and coefficients  $K_i$  and  $B_0$ , and in that the calculation means (7) are arranged to calculate a value  $V_p$  of the characteristic parameter from values of the luminescence spectral density  $L_i$  of the said continuous spectrum obtained for the N spectral bands  $\sigma_i$ , according to the formula:

$$V_p = B_0 + \sum_{i=1}^N K_i L_i$$

45. A device according to claim 44, *characterised in* that N is a number between 5 and 30.

10 46 A device according to claim 35, *characterised in* that the group of characteristic parameters further comprises alcoholic strength; total sugar content; total acidity; pH; concentration of acetic acid; concentration of malic acid; concentration of tartaric acid; concentration of lactic acid; and content of phenolic compounds.

METHOD AND DEVICE FOR OBJECTIVE QUALITATIVE ANALYSIS OF GRAPE MUSTS  
AND/OR WINES BY BROADBAND INFRA-RED SPECTROMETRY

The invention relates to a method and a device for objective qualitative analysis by broadband infrared spectrometry of grape musts intended for vinification and/or wines, collectively referred to in this text as "vinification compositions".

For a long time a solution has been sought that would permit an objective and rapid qualitative evaluation of grape musts intended for vinification. Such an evaluation would make it possible to determine objectively the price of a grape harvest according to its quality. Furthermore, it would be possible to better select the musts according to the qualities of the desired wines, and ultimately to employ vinification technologies that are better adapted to the quality of the grape musts. To be effective this objective analysis must be able to be carried out very quickly (within no more than about 1 to 2 minutes) on the raw musts at the vinification sites (and not only in a laboratory).

Until now the musts have been evaluated in a coarse manner, essentially by measuring the content of sugar by refractometry or densitometry. In rare cases this measurement has been supplemented by measuring the total acidity and the pH by traditional chemical titration methods. Sometimes a subjective semi-quantitative evaluation of the presence of laccase, (an enzyme secreted by *Botrytis cinerea*, a grape parasite) is carried out. Nevertheless, as this enzyme itself is destroyed by the products of the reactions that it catalyses, its measurement is not useful.

20 The same problem arises for the objective qualitative analysis of wines that would permit an objective determination of their value and the qualities.

It has been proposed to determine the alcoholic strength of wines by using a spectrophotometer in the near infrared range for 19 distinct wavelengths ("l'infraalyzer 400 : une determination automatique du titre alcoometrique des vins" CABANIS et al, Fran...Oenol 89, 75-79, 1983).

25 Nevertheless, the use of such an apparatus is long and complex, only provides results for the measurement of ethanol and sugar reducing agents, and does not permit a complete objective analysis of the quality of the musts and/or the wines.

For more than 20 years, various other theoretical methods of analysis of grape musts and/or wines have been described, but none of them has become the object of practical exploitation. They are 30 much too complex to put into practice and/or do not provide complete and reproducible objective results.

The invention aims to remedy these drawbacks by proposing a method and a device permitting the provision of an objective qualitative analysis of grape musts and/or wines and capable of being put into practice outside a laboratory –particularly at a vinification site.

The invention aims then to permit this analysis to be obtained in a rapid manner, particularly 35 within a maximum duration of 1 to 2 minutes.

The invention also permits this analysis to be obtained in a simple automatic manner, in a single analysis step performed automatically, without it being necessary to carry out chemical preparations, manipulations or adjustments.

The invention also aims to permit reliable and complete analyses to be obtained permitting an 5 objective appreciation of the quality of a wine and/or must and/or the harvest from which a must originates, in a manner particularly enabling an objective determination of its price and facilitating the defining of the subsequent measures to be taken for the vinification and/or the conservation and/or the commercialisation.

To achieve this, the invention concerns a method for the objective qualitative analysis of liquid 10 vinification compositions, characterised in that

- an initial configuration and calibration step (13) is carried out during which:
  - a group of characteristic parameters are selected that are capable of characterising the quality of a vinification composition to be analysed, and comprising the concentration levels in the vinification composition of characteristic compounds chosen among at least a compound produced by 15 *Botrytis cinerea*, at least a compound produced by yeasts, at least a compound produced by acetic bacteria, and at least a compound produced by lactic acid bacteria,
  - calibration values of spectroscopic criteria are determined and recorded for each characteristic parameter, the spectroscopic criteria being chosen to enable evaluation of the characteristic parameter in a vinification composition from an infrared absorption spectrum, which can 20 be generated on a sample of this vinification composition,
- for each vinification composition to be analysed:
  - a spectroscopic analysis is carried out during which a continuous Infrared absorption spectrum of a sample of this vinification composition is generated,
  - the spectroscopic criteria of each characteristic parameter are applied to the said 25 continuous spectrum in such a way that the value of this characteristic parameter in the vinification composition is evaluated by automatic calculation.

Advantageously and according to the invention, the group of characteristic parameters comprises the concentration of each of the characteristic compounds belonging to the group consisting of gluconic acid, ethanal, ethyl acetate, arabitol, mannitol, sorbitol, 2,3-butanediol, methyl-3 butanol-1, glycerol, 30 mesoinositol and isoamyl acetate. Thus calibration values are determined and recorded for these characteristic parameters, which are evaluated by calculation for each vinification composition to be analysed.

Advantageously and according to the invention, the spectroscopic criteria are chosen to enable evaluation of each characteristic parameter in a vinification composition from a near and mid infrared 35 absorption spectrum, and the near and mid infrared continuous spectrum of each vinification composition to be analysed is generated.

Advantageously and according to the invention, the continuous spectrum is generated by Fourier transform interferometric spectroscopy.

Advantageously and according to the invention, at least an objective quality index of the vinification composition is calculated automatically as a function of the said value evaluated from at least 5 a characteristic parameter for the vinification composition to be analysed. Advantageously and according to the invention the function is a polynomial function.

Advantageously and according to the invention the following are calculated:

- a first quality index, representing the attack on the harvest by *Botrytis cinerea*, at least as a function of the value of concentration levels of gluconic acid, mannitol and sorbitol evaluated from the 10 continuous spectrum by application of spectroscopic criteria,

- a second quality index, representing the attack by yeasts, at least as a function of the value of the concentration levels of ethanal, ethyl acetate, arabitol, 2,3-butanediol, methyl-3 butanol-1, glycerol, and isoamyl acetate, evaluated from the continuous spectrum by application of spectroscopic criteria,

- a third quality index representing the attack by acetic bacteria, at least as a function of values of 15 the concentration levels of acetic acid, ethyl acetate and of 2,3-butanediol evaluated from the continuous spectrum obtained by application of spectroscopic criteria,

- a fourth quality index representing the attack by lactic acid bacteria, at least as function of the value of concentration levels of lactic acid, mannitol and of 2,3-butanediol, evaluated from the continuous spectrum by application of spectroscopic criteria,

20 - and a fifth quality index representing the fermentation ability, at least as a function of the value of the concentration level of mesoinositol.

Advantageously and according to the invention each characteristic parameter is evaluated and each quality index is calculated by data processing immediately after the spectroscopic analysis step has been carried out.

25 Advantageously and according to the invention, the spectroscopic criteria comprise for each characteristic parameter, for a selection of an integral number N of spectral bands  $\sigma_i$  of predetermined wavelengths, the value of the luminescence spectral density  $L_i$  of the said continuous spectrum, and a value  $V_p$  of the characteristic parameter is calculated according to the formula:

$$V_p = Bo + \sum_{i=1}^N Ki L_i$$

30

where  $Bo$  and  $Ki$  are predetermined coefficients. Advantageously and according to the invention, N is included in the group from 5 to 30 - particularly in the order 15 -.

Advantageously and according to the invention the group of characteristic parameters further comprises the alcoholic volumetric strength; total sugar content; total acidity; pH; concentration of acetic 35 acid; concentration of malic acid; concentration of tartaric acid, concentration of lactic acid, and content

of phenolic compounds. In this way the calibration values are determined and recorded for the characteristic parameters which are evaluated by calculation for each vinification composition to be analysed. These characteristic parameters may then also be used and evaluated to determine one or more quality indices.

5 The invention also extends to a device for carrying out a method according to the invention.

Accordingly, the invention also concerns a device for objective qualitative analysis of liquid vinification compositions, characterised in that it comprises:

- memory means wherein calibration values of spectroscopic criteria are recorded for a group of characteristic parameters capable of characterising the quality of a vinification composition to be analysed, the group comprising the concentration levels in the vinification composition of characteristic compounds selected among at least a compound produced by *Botrytis cinerea*, at least a compound produced by yeasts, at least a compound produced by acetic bacteria, and at least a compound produced by lactic acid bacteria, the said spectroscopic criteria being selected for each characteristic parameter to enable evaluation of the characteristic parameter in a vinification composition from an infrared absorption spectrum that can be generated on a sample of this vinification composition,

10 spectroscopic analysis means for collecting a vinification composition sample and generating an infrared continuous spectrum of this sample,

15 calculation means arranged to apply the spectroscopic criteria of each characteristic parameter to the said continuous spectrum and to evaluate by automatic calculation the value of this characteristic parameter in the vinification composition.

20 Advantageously and according to the invention, the group of characteristic parameters comprises the concentration of each of the characteristic compounds belonging to the group consisting of gluconic acid, ethanal, ethyl acetate, arabitol, mannitol, sorbitol, 2,3-butanediol, methyl-3butanol-1, glycerol, mesoinositol and isoamyl acetate. Then calibration values of these concentration levels are recorded in the memory means and the calculation means are arranged to evaluate the value of these characteristic parameters.

25 Advantageously and according to the invention, the selection criteria are chosen for each characteristic parameter to enable evaluation of the characteristic parameter in a vinification composition from a near and mid infrared spectrum, and the means for spectroscopic analysis are arranged to generate the near and mid infrared continuous absorption spectrum. Advantageously and according to the invention, the means for spectroscopic analysis comprise a Fourier transform interferometric spectrometer.

30 Advantageously and according to the invention, the calculation means are arranged to calculate automatically at least an objective quality index of the vinification composition as a function of said value evaluated from at least a characteristic parameter for the vinification composition to be analysed. Advantageously and according to the invention, the function is a polynomial function.

35 Advantageously and according to the invention the calculation means are arranged to calculate:

- a first quality index, representing the attack on the harvest by *Botrytis cinerea*, at least as a function of the value of concentration levels of gluconic acid, mannitol and sorbitol evaluated from the continuous spectrum by application of spectroscopic criteria,
- a second quality index, representing the attack by yeasts, at least as a function of the value of 5 the concentration levels of ethanal, ethyl acetate, arabitol, of 2,3-butanediol, of methyl-3 butanol-1, of glycerol, and of isoamyl acetate evaluated from the continuous spectrum by application of spectroscopic criteria,
- a third quality index representing the attack by acetic bacteria, at least as a function of values of the concentration levels of acetic acid, ethyl acetate and of 2,3-butanediol evaluated from the 10 continuous spectrum obtained by application of spectroscopic criteria,
- a fourth quality index representing the attack by lactic acid bacteria, at least as function of the value of concentration levels of lactic acid, mannitol and of 2,3-butanediol, evaluated from the continuous spectrum by application of spectroscopic criteria,
- and a fifth quality index representing the fermentation ability, at least as a function of the value 15 of the concentration level of mesoinositol.

Advantageously and according to the invention, the calculation means are arranged to evaluate each characteristic parameter and to calculate each quality index by data processing immediately after the generation of the continuous spectrum by the spectroscopic analysis means, and to provide the results of these calculations to means that are read by a user. Advantageously and according to the 20 invention, the means that are read comprise means for printing a report of results.

Advantageously a device according to the invention, is characterised in that for each characteristic parameter, the calibration values comprise an integral number N of spectral bands  $\omega_i$  of predetermined wavelengths, and coefficients  $K_i$  and  $B_0$ , and in that calculation means are arranged to calculate a value  $V_p$  of the characteristic parameter from the values of the luminescence spectral density 25  $L_i$  of the said continuous spectrum obtained for the N spectral bands  $\omega_i$  according to the formula,

$$V_p = B_0 + \sum_{i=1}^N K_i L_i$$

Advantageously and according to the invention N lies between 5 and 30, particularly in the order of 15.

Advantageously and according to the invention, the group of characteristic parameters further comprises the volumetric alcoholic strength; total sugar content; total acidity; pH; concentration of acetic 30 acid; concentration of malic acid; concentration of tartaric acid; concentration of lactic acid, and content of phenolic compounds. Calibration values of these concentration levels are then recorded in the memory means and the calculation means are arranged to evaluate the value of the characteristic parameters

The invention also concerns a method and a device in combination with all or with part of the 35 above or below mentioned characteristics.

In a method according to the invention, the initial configuration and calibration step is carried out once and for all, e.g. in the factory when manufacturing the device according to the invention. Afterwards, the analysis of each vinification composition is obtained in a single automatic, simple and rapid step.

5 Accordingly, the invention permits one to obtain an analysis of a vinification composition in a simple, rapid, reliable objective and automatic manner. In particular, it should be noted that the inventors have found that among the multitude of different compounds figuring in the composition of the vinification compositions, the characteristic parameters chosen in a method according to the invention are specific to a quality index, and can be measured in practice by infrared spectroscopy from a 10 continuous spectrum in a meaningful and useful manner, particularly by Fourier transform interferometric spectrometry.

The inventor has also found that this technology, even though on the face of it, it is considered one of the most complex within the area of spectrometry, particularly as it normally requires the carrying out of calibrations, adjustments, and heavy and complex calculations, can in fact provide rapid and 15 precise results when it is applied to a vinification composition. Particularly, even though, on the face of it, it appears much more burdensome and complex than the use of the previously proposed spectrometer with 19 distinct wavelengths, on the contrary, the invention has proved able to permit, after configuration and calibration, a complete objective reliable and rapid analysis to be obtained. Further, the inventor has found that it is possible to provide the results directly in synthesised and simplified form in a few indices 20 of quality that easily can be interpreted by vinification professionals.

Other objects advantages and characteristics of the invention will appear from the reading of the examples in the following description with reference to the drawings in which:

Figure 1 is a schematic diagram of a device according to the invention,

Figure 2 is a flow diagram of a method according to the invention.

25 Figure 1 illustrates a device according to the invention comprising a needle 1 for extraction of a liquid vinification composition sample for which an objective qualitative analysis is desired. This extraction needle 1 is connected to filter means 2 and to means 3 for pumping a predetermined quantity of vinification composition forming the sample to be analysed, permitting the introduction of this sample into a spectroscopic analysis cell 4. The pumping means 3 are motorised and automatically controlled to 30 extract the predetermined quantity of liquid vinification composition and maintain it in the cell 4 for a period of time to permit the generation of a continuous infrared absorption spectrum of the sample in the cell 4. These pumping means 3 can be formed in any known manner from an automatic control device, one or more electric motors and pumps, particularly peristaltic pumps. A pump can be provided upstream of the cell 4 to introduce the sample into this cell 4, and another pump can be provided 35 downstream from the cell 4 to remove the sample from the cell 4 after analysis and move it towards a discharge outlet 5 as shown in Figure 1.

Also the device according to the invention comprises a Fourier transform interferometric spectrometer 6 arranged to generate a continuous infrared absorption spectrum – particularly in the

near or mid infrared range, i.e. for wavelengths comprising 800 nm and 15000 nm – of the sample in the cell 4. Such a Fourier transform interferometric spectrometer providing a continuous near or mid infrared absorption spectrum is known as such. In particular, the spectrometer FT 120 sold by FOSS FRANCE SA (Nanterre, France) can be cited. Such an interferometer is fully automatic and incorporates means 5 for calculating the continuous spectrum by Fourier transformation from the generated interferogram, and software for using the data. Furthermore, these calculation means are arranged to provide the results of the continuous spectrum in the form of numerical data, which can be input directly into the microprocessor data processing means 7 of the device according to the invention.

The data processing means 7 are arranged and programmed to carry out the different 10 calculations of a method according to the invention. A mass memory 8, such as a hard disk and/or a disk or diskette reader is associated with the data calculation means 7, which also comprises the assembly of electronic components and the various customary peripherals which are necessary for their operation and which are not shown in Figure 1.

In the mass memory 8 are recorded the calibration values of spectroscopic criteria for a group of 15 characteristic parameters characterising the quality of a vinification composition to be analysed, this group comprising the concentration in the vinification composition of characteristic compounds chosen among at least a compound produced by *Botrytis cinerea*, at least a compound produced by yeasts, and at least a compound produced by acetic bacteria, at least a compound produced by lactic acid bacteria, the said spectroscopic criteria being selected for each characteristic parameter to permit evaluation of 20 the characteristic parameter in a vinification composition from an infrared absorption spectrum which can be generated on a sample of the vinification composition.

Furthermore, the data processing means 7 are arranged to apply each spectroscopic criterion of each characteristic parameter to the continuous spectrum produced by the interferometer spectrometer 6 and to evaluate by automatic calculation the value of the characteristic parameter in the vinification 25 composition located in the cell 4.

In particular for each characteristic parameter the calibration values stored in the mass memory 8 comprise coefficients  $K_i$  to be applied to the luminescence spectral density values  $L_i$  of the continuous spectrum obtained for different spectral bands  $\omega_i$  of predetermined wavelengths. And the data processing means 7 calculate the value  $V_p$  of the characteristic parameter according to the formula:

$$30 \quad V_p = B_0 + \sum_{i=1}^N K_i L_i$$

where  $N$  is the integral number of retained spectral bands, which preferably lies between 5 and 30, in particular in the order of 15.

The various calibration values are established for each characteristic parameter from a statistic analysis of a large number of known reference samples from vinification compositions in which the real 35 value of the characteristic parameter is known, e.g. predetermined by measured amounts added or physical-chemical measurement in a laboratory. The statistical correlation between the continuous spectra obtained for the various vinification compositions and the known real values of each

characteristic parameter allowing a determination of the various coefficients  $K_i$ , can be determined from a statistical calculation made by means of a known statistical calculation program, in particular a statistical spectroscopic program such as the one marketed by the company FOSS FRANCE SA (Nanterre, France).

5 It should be noted that the inventor has determined that from a certain number of vinification composition reference samples originating from principal grape vines (particularly Chardonnay, Cabernet, Sauvignon, Merlot, Carignan, Syrah, ...) and having different sanitary and maturity qualities, an almost universal calibration can be obtained enabling evaluation of characteristic parameters for all vinification compositions with a good reliability.

10 But the choice, the number, and the characteristics (origin, grape vines, vinification methods, ...) of the vinification composition reference samples can also be arranged, according to the same characteristics (origin, grape vines, vinification method, ...) of the vinification compositions to be analysed for optimising the calibration. For example, it is possible to supplement the initial calibration with reference samples from vinification compositions provided from the same wine-growing region as the 15 vinification compositions that have to be analysed.

The calculation means 7 are also arranged to calculate automatically at least one objective quality index of the vinification composition as a function of the value of at least one characteristic parameter. More particularly according to the invention, the calculation means 7 are arranged to calculate:

20 - a first quality index  $Q_1$ , representing the attack of the harvest by *Botrytis cinerea*, at least as a function of the value of concentration levels of gluconic acid, mannitol and sorbitol evaluated from the continuous spectrum by application of spectroscopic criteria,

- a second quality index  $Q_2$ , representing the attack by yeasts, at least as a function of the value of the concentration levels of ethanal, ethyl acetate, arabitol, of 2,3-butanediol, of methyl-3 butanol-1, of 25 glycerol, and of isoamyl acetate evaluated from the continuous spectrum by application of spectroscopic criteria,

- a third quality index  $Q_3$  representing the attack by acetic bacteria, at least as a function of values of the concentration levels of acetic acid, ethyl acetate and of 2,3-butanediol evaluated from the continuous spectrum obtained by application of spectroscopic criteria,

30 - a fourth quality index  $Q_4$  representing the attack by lactic acid bacteria, at least as function of the value of concentration levels of lactic acid, mannitol and of 2,3-butanediol, evaluated from the continuous spectrum obtained by application of spectroscopic criteria,

- and a fifth quality index  $Q_5$  representing the fermentation ability, is calculated at least as a function of the value of the concentration of mesoinositol.

35 More particularly, each quality index  $Q_i$  is calculated as a polynomial function of various characteristic parameters. Furthermore, one or more other general quality indices can be established from the volumetric alcoholic strength, total sugar content, total acid, pH, concentration of malic acid and tartaric acid, and the content of phenolic compounds.

The calculation means 7 are advantageously arranged to evaluate each characteristic parameter and to calculate automatically each quality index  $Q_j$  by data processing, and this is done immediately after the generation of the continuous spectrum by the interferometric spectrometer 6.

The choice of the various quality indices  $Q_j$  and their calculation formulas from the values of the 5 characteristic parameters can vary to a certain degree according to the nature of the vinification composition (must and/or wine) or according to the characteristics (origin, grape vine, vinification methods ...) of the vinification compositions to be analysed.

Nevertheless, the inventors have determined that in practice the pertinent calibration values of the various above-mentioned characteristic parameters can be defined by an integral number  $N$  of 10 wavelengths lying between 5 and 30, particularly in the order of 15 – permitting calculation of the same quality indices  $Q_j$  by the same calculation formulas which remain valid for the majority of musts and wines that can be encountered.

In practice, the general character of the calculations depends on the number of vinification composition samples which were used to establish by statistical methods the calibration values, and of 15 the sophistication of the spectroscopic criteria employed, i.e. in particular the number of wavelengths used for each parameter. This number of wavelengths  $N$  can vary from one parameter to another, or can, on the contrary – be the same for all the characteristic parameters. The higher it is, the longer are the subsequent data processing calculations to be carried out on the continuous spectrum.

With the actual data means it has been determined that for a number  $N = 15$ , for the various 20 characteristic parameters mentioned above, it is possible to obtain results of quality indices in less than one minute.

The various calibration values  $K_i$  and  $B_i$  may be statistically determined either by a multiple linear regression or preferably, by a PLS calculation ("Partial least square"). The calibration values may also comprise corrections of slope and ordinate at the initial origin, i.e. constants  $\alpha$  and  $\beta$  to be 25 applied to the above mentioned value  $V_p$ , according to each characteristic parameter, according to the formula  $\alpha \cdot V_p + \beta$ , wherein these constants will vary from one device to another, particularly to compensate for the respective drifts for each interferometric spectrometer 6. The quality indices are thus calculated from the corrected values  $\alpha \cdot V_p + \beta$ .

The results of the analysis are communicated by the data processing means 7 to a user interface 30 9 comprising reading means 10 including means for printing a report with results and /or a display screen. Preferably, the results are communicated in the form of a list of the various calculated quality indices  $Q_j$ . Preferably, the report also shows the date and/or time of the analysis and identification information for the analysed vinification composition sample. Advantageously, the user interface 9 also comprises a control button 11 permitting initiation of the extraction of a sample through the needle 1, 35 spectroscopic analysis of the sample, and the calculation of the quality indices.

The device according to the invention is particularly compact, simple and reliable to use. Particularly it should be noted that all of the above-mentioned units except the extraction needle 1 and the user interface 9 can be integrated inside a closed casing 12. The user only has to locate the

extraction needle 1 in the sample contained in a container, and then operate the control button 11. After a wait of a few seconds, in the order of one to two minutes at the most, he will receive the results from the reading means 10 in the form of a list of quality indices.

Figure 2 represents a flow diagram of an analysis method according to the invention. The 5 method comprises an initial configuration and calibration step 13 during which the device according to the invention is prepared to enable its operation. In this initial step 13, the various characteristic parameters used to characterise the quality of a vinification composition to be analysed later are first chosen. These characteristic parameters are those that were mentioned above. For each characteristic parameter, the N spectral bands  $\omega_i$  are chosen, and by statistical calculations the various calibration 10 values  $K_i$ ,  $B_i$  for the various spectral bands  $\omega_i$  are determined, and this is done by analysing a multitude of vinification compositions for which the values of the characteristic parameters are known by other means. This initial statistical calculation is carried out during step 14. In the following step 15 the various calibration values  $i$ ,  $K_i$  and  $B_i$  are stored in the mass memory 8. Once this initial configuration and calibration step 13 has been performed, the device is ready to function, i.e. it is ready to carry out the 15 analysis of a vinification composition in step 16. In this analysis step 16, at first, a sample extraction 17 is carried out, and then the continuous spectrum of this sample is established 18 with the aid of the Fourier transform interferometer spectrometer 6. From this spectrum and the previously stored calibration values  $\omega_i$ ,  $K_i$  and  $B_i$ , the various values  $V_p$  of the various characteristic parameters are calculated in step 19 for the various spectral bands  $\omega_i$ . Then in step 20 the various quality indices  $Q_i$  are 20 calculated from the various values  $V_p$  of the characteristic parameters, and these quality indices are delivered to the reading means 10.

Example:

For each characteristic parameter a series of several samples is generated from musts or wines by adding measured amounts of the compound corresponding to this characteristic parameter. The 25 generated samples then serve to provide the initial configuration and calibration step for a device according to the invention. The various spectroscopic analyses are performed by the interferometer spectrometer FT 120 from the company FOSS FRANCE SA, and the statistical calculations are performed by the spectral statistical analysis programs sold with this device.

The following tables show wavelengths (by pin number) defining the spectral bands  $i$  and the 30 calibration coefficients  $K_i$  and  $B_i$  for the spectral bands  $i$  – obtained from the samples with measured added amounts for each characteristic parameter. Also the coefficients and of correction corresponding to the apparatus used are given.

The tables also show the discrimination rate of concentration levels obtained with the number of spectral bands  $\omega_i$  used.

35 Furthermore, the same samples are used to re-evaluate according to the invention their respective concentration levels in compounds corresponding to the characteristic parameter. The tests are repeated. The values obtained are compared to values corresponding to the measured added

amounts provided in each sample, which are theoretical values. With these values is obtained a calculated standard deviation ETC, and a coefficient of correlation  $R^2$  also given in each table.

The same tests are performed in similar manner for ethanal, arabitol, methyl-3 butanol-1, isoamyl acetate, glycerol, 2,3 butanediol, ethyl acetate, mannitol, sorbitol, gluconic acid, and 5 mesoinositol.

The same results can also be obtained with lactic acid, malic acid, tartaric acid, glucose and fructose, total acidity, pH, acetic acid, the total alcoholic content, and the content of phenolic compounds.

ETHANAL				
$\sigma_i$	DE	A	$K_i$	
$\sigma 1$	445	445	K 1	102675.16746
$\sigma 2$	451	451	K 2	-123721.54770
$\sigma 3$	375	375	K 3	- 42146.44657
$\sigma 4$	359	360	K 4	- 14287.71152
$\sigma 5$	308	309	K 5	- 28381.96828
$\sigma 6$	294	295	K 6	83899.61893
$\sigma 7$	557	557	K 7	42104.33149
$\sigma 8$	263	272	K 8	- 16677.82213
$\sigma 9$	400	400	K 9	32694.87756
$\sigma 10$	283	284	K 10	25078.21399
$\sigma 11$	500	503	K 11	54137.67301
$\sigma 12$	390	394	K 12	- 21740.48588
$\sigma 13$	742	742	K 13	14944.19251
$\sigma 14$	299	300	K 14	- 41194.49975

Discrimination rate = 97.78 %  
 $Bo = -830.25865$   
 $\alpha = 1$   
 $\beta = 0$   
Number of samples = 89  
Concentration varying from 0 to 1000 mg/l  
ETC = 38.4492  
 $R^2 = 0.9822$

The ethanal not originally present in the musts is specifically formed by the yeasts. This  
5 permits the presence of yeasts and the start of fermentation to be detected and can be used in the quality index  $Q_2$ .

## ARABITOL

σi	DE	A	Ki	
σ 1	445	446	K 1	34011.16421
σ 2	374	374	K 2	- 51783.46396
σ 3	448	457	K 3	- 122987.87178
σ 4	358	358	K 4	- 93391.60145
σ 5	353	353	K 5	32028.99373
σ 6	307	307	K 6	13702.96397
σ 7	295	295	K 7	53867.88537
σ 8	562	563	K 8	36605.77772
σ 9	301	302	K 9	- 81872.88535
σ 10	330	332	K 10	105552.06113
σ 11	400	400	K 11	46749.66484
σ 12	269	269	K 12	- 5973.89318
σ 13	393	393	K 13	- 39254.22277
σ 14	382	382	K 14	78489.74000
σ 15	377	378	K 15	- 53786.09915
σ 16	768	768	K 16	4573.32521
σ 17	250	252	K 17	3159.98168
σ 18	290	290	K 18	13594.96085
σ 19	384	384	K 19	- 46051.52112
σ 20	371	371	K 20	45257.05498

Discrimination rate = 94.21 %

$B_0 = 23.67991$

$\alpha = 1,0006$

$\beta = 0,4956$

Number of samples = 89

Concentration varying from 0 to 350 mg/l

ETC = 19.7440

$R^2 = 0.9593$

Arabitol has the same properties as ethanal, and can be used in the calculation of the quality index  $Q_2$ .

## METHYL -3 - BUTANOL 1

$\sigma i$	DE	A	Ki	
$\sigma 1$	445	445	K 1	37001.13793
$\sigma 2$	451	451	K 2	- 31825.73760
$\sigma 3$	740	740	K 3	- 11537.90703
$\sigma 4$	359	360	K 4	2750.26865
$\sigma 5$	294	294	K 5	27669.36454
$\sigma 6$	309	309	K 6	- 10792.87694
$\sigma 7$	566	568	K 7	12235.25346
$\sigma 8$	379	379	K 8	- 2020.95509
$\sigma 9$	271	271	K 9	- 3134.44842
$\sigma 10$	393	393	K 10	- 8172.41484
$\sigma 11$	399	400	K 11	- 6272.69448
$\sigma 12$	502	502	K 12	16729.91741
$\sigma 13$	265	266	K 13	- 1885.60801
$\sigma 14$	283	283	K 14	6328.39641
$\sigma 15$	326	326	K 15	- 22107.92011

Discrimination rate= 97.28%

 $Bo = - 308.18771$  $\alpha = 1$  $\beta = 0$ 

Number of samples = 96

Concentration varying from 0 to 400 mg/l

ETC = 19.4254

 $R^2 = 0.9714$ 

Methyl - 3 - butanol 1 has substantially the same properties as ethanal, and may be used in the calculation of the quality index  $Q_2$ .

ISOAMYL ACETATE				
σi	DE	A	Ki	
σ 1	445	446	K 1	8869.55722
σ 2	351	351	K 2	-4724.52417
σ 3	331	331	K 3	16284.72853
σ 4	358	358	K 4	- 13187.74050
σ 5	451	451	K 5	- 18796.27131
σ 6	378	379	K 6	- 18948.30736
σ 7	294	295	K 7	13557.03424
σ 8	567	567	K 8	- 8980.89314
σ 9	301	302	K 9	- 13349.49570
σ 10	308	309	K 10	2551.18673
σ 11	398	398	K 11	- 7997.38660
σ 12	683	683	K 12	- 2168.91044
σ 13	385	385	K 13	2529.67798
σ 14	393	393	K 14	- 10198.64556
σ 15	281	281	K 15	- 1074.13624
σ 16	354	354	K 16	12906.05284
σ 17	270	270	K 17	- 822.14254
σ 18	253	253	K 18	2550.37446
σ 19	256	256	K 19	- 2670.03048
σ 20	250	250	K 20	1499.44706

Discrimination rate= 90.67 %

Bo = 93.23514

$\alpha$  = 1

$\beta$  = 0

Number of samples = 98

Concentration varying from 0 to 100 mg/l

ETC = 8.0368

R<sup>2</sup> = 0.9185

Isoamyl acetate has the same properties as ethanal, and may be used in the calculation of the quality index Q<sub>2</sub>

GLYCEROL				
$\sigma i$	DE	A	Ki	
$\sigma 1$	447	447	K 1	- 51.35261
$\sigma 2$	302	302	K 2	- 50.58692
$\sigma 3$	366	366	K 3	13.52560
$\sigma 4$	353	353	K 4	8.17399
$\sigma 5$	393	396	K 5	30.39023
$\sigma 6$	741	742	K 6	49.85179
$\sigma 7$	383	384	K 7	110.81504
$\sigma 8$	390	390	K 8	254.95298
$\sigma 9$	334	334	K 9	141.55803
$\sigma 10$	361	362	K 10	211.46653
$\sigma 11$	371	371	K 11	219.53447
$\sigma 12$	274	276	K 12	- 28.56856
$\sigma 13$	769	769	K 13	- 20.34421
$\sigma 14$	392	392	K 14	- 89.82594
$\sigma 15$	450	450	K 15	21.01462

Discrimination rate = 97.40%

$Bo = - 2.32923$

$\alpha = 1$

$\beta = 0$

Number of samples = 55

Concentration varying from 0 to 25 000 mg/l

ETC = 0.0580

$R^2 = 0.9728$

2,3 - BUTANEDIOL				
σi	DE	A	Ki	
σ 1	446	446	K 1	- 61624.13783
σ 2	566	567	K 2	115022.21030
σ 3	358	358	K 3	315082.03317
σ 4	295	295	K 4	- 25522.71367
σ 5	311	311	K 5	- 61376.66922
σ 6	352	354	K 6	- 188041.60059
σ 7	450	452	K 7	108380.73874
σ 8	400	400	K 8	- 59989.91275
σ 9	738	739	K 9	- 99409.32842
σ 10	378	379	K 10	256606.18712
σ 11	264	264	K 11	- 31295.02573
σ 12	258	260	K 12	15678.84745
σ 13	320	321	K 13	- 165106.31604
σ 14	331	333	K 14	121745.91275
σ 15	398	398	K 15	11576.64077

Discrimination rate = 98.67%  
 Bo = 354.21365  
 $\alpha = 1$   
 $\beta = 0$   
 Number of samples = 96  
 Concentration varying from 333 to 1350 mg/l  
 ETC = 74.6002  
 $R^2 = 0.9861$

5 The yeasts and the bacteria produce the 2,3 - butanediol. It can be used in the calculation of the quality indices Q2, Q3 and Q4.

ETHYL ACETATE				
σi	DE	A	Ki	
σ 1	445	445	K 1	- 4022.10920
σ 2	450	450	K 2	- 65335.82429
σ 3	740	740	K 3	29242.22311
σ 4	358	358	K 4	10502.82006
σ 5	301	301	K 5	-108382.38049
σ 6	399	399	K 6	19933.58344
σ 7	308	308	K 7	41565.01236
σ 8	329	330	K 8	177242.60253
σ 9	295	295	K 9	33912.39645
σ 10	391	391	K 10	58902.89916
σ 11	260	260	K 11	24959.20375
σ 12	505	505	K 12	-100795.64524
σ 13	566	566	K 13	41291.76755
σ 14	269	270	K 14	-3775.18718
σ 15	397	397	K 15	-55403.16130
σ 16	377	378	K 16	-78470.10709
σ 17	382	382	K 17	69763.39685
σ 18	343	345	K 18	-102702.52928
σ 19	282	282	K 19	-3911.36127
σ 20	770	770	K 20	-10660.03193

Discrimination rate = 96.11 %

$B_0 = 1302.08571$

$\alpha = 1$

$\beta = 0$

Number of samples = 98

Concentration varying from 0 to 500 mg/l

ETC = 26.9794

$R^2 = 0.9608$

The Ethyl acetate initially not present in the musts is formed specifically by the acid bacteria and certain yeasts. It may be used in the calculation of the quality indices  $Q_2$  and  $Q_3$ .

## MANNITOL

σi	DE	A	Ki	
σ 1	446	446	K 1	-3179.89056
σ 2	566	567	K 2	5001.38528
σ 3	358	358	K 3	15593.50139
σ 4	295	295	K 4	-1886.50039
σ 5	311	311	K 5	-3637.02250
σ 6	352	354	K 6	-10007.26393
σ 7	451	451	K 7	5416.46202
σ 8	400	400	K 8	-2785.76190
σ 9	738	739	K 9	-6745.93770
σ 10	378	379	K 10	12399.78063
σ 11	264	264	K 11	-1173.32860
σ 12	257	260	K 12	919.95926
σ 13	321	321	K 13	-7657.43606
σ 14	331	332	K 14	6569.68872
σ 15	762	763	K 15	1477.56746

Discrimination rate = 98.70%

Bo = 52.25987

 $\alpha = 1$  $\beta = 0$ 

Number of samples = 98

Concentration varying from 90 to 750 mg/l

ETC = 3.7352

 $R^2 = 0.9859$ 

5 The mannitol is specifically produced by the lactic acid bacteria and *Botrytis cinerea*. It can be used to calculate Q1 and Q2.

SORBITOL				
σi	DE	A	Ki	
σ 1	1365	1365	K 1	-170763.92687
σ 2	1724	1724	K 2	-74236.87161
σ 3	1516	1520	K 3	17284.26311
σ 4	1388	1388	K 4	160555.50077
σ 5	1165	1165	K 5	-285759.08492
σ 6	1986	1986	K 6	25471.99288
σ 7	1404	1415	K 7	286285.44757
σ 8	1057	1057	K 8	11876.72404
σ 9	2881	2881	K 9	-505.86779
σ 10	1037	1037	K 10	-10572.17495
σ 11	1446	1446	K 11	-152242.11656
σ 12	1222	1222	K 12	180946.67117
σ 13	1496	1496	K 13	134293.16678
σ 14	1523	1527	K 14	-137429.76254
σ 15	964	964	K 15	6861.61554

Discrimination rate= 88.50%  
 Bo = 2852.12225  
 $\alpha = 0.9993$   
 $\beta = 0.6461$   
 Number of samples = 50  
 Concentration varying from 30 to 300 mg/l  
 ETC = 22.3337  
 $R^2 = 0.9167$

5 The sorbitol is specifically produced by *Botrytis cinerea*. It can be used to calculate the quality index Q2.

GLUCONIC ACID				
σi	DE	A	Ki	
σ 1	449	449	K 1	27.59437
σ 2	352	352	K 2	-494.94507
σ 3	333	333	K 3	354.10721
σ 4	294	294	K 4	367.10310
σ 5	302	302	K 5	-157.88863
σ 6	396	397	K 6	-276.03158
σ 7	390	391	K 7	-115.88962
σ 8	365	370	K 8	2.66976
σ 9	377	377	K 9	140.25803
σ 10	267	268	K 10	27.71803
σ 11	384	384	K 11	51.07514
σ 12	741	741	K 12	-223.32023
σ 13	261	261	K 13	-67.28365
σ 14	372	372	K 14	-29.43258
σ 15	364	364	K 15	-63.25036

Discrimination rate = 99.44 %  
 $Bo = 3.47339$   
 $\alpha = 1$   
 $\beta = 0$   
Number of samples = 98  
Concentration varying from 0 to 8000 mg/l  
ETC = 0.1692  
 $R^2 = 0.9955$

5 The gluconic acid is specifically produced by *Botrytis cinerea* and can be used in the calculation of the quality index Q1.

MESOINOSITOL				
σi	DE	A	Ki	
σ 1	445	446	K 1	-51313.80771
σ 2	450	450	K 2	-42575.49224
σ 3	740	740	K 3	-52662.13173
σ 4	358	358	K 4	-21329.77085
σ 5	301	301	K 5	-175911.52518
σ 6	399	399	K 6	-91799.02350
σ 7	308	308	K 7	24446.66022
σ 8	567	567	K 8	-3575.37646
σ 9	331	331	K 9	247459.08477
σ 10	295	295	K 10	43222.40578
σ 11	390	390	K 11	51560.39057
σ 12	505	505	K 12	-127663.26606
σ 13	260	260	K 13	49854.22008
σ 14	269	269	K 14	-11978.62572
σ 15	377	378	K 15	-59824.90209

Discrimination rate= 94.25%

Bo = 3246.66966

$\alpha = 1$

$\beta = 0$

Number of samples = 96

Concentration varying from 220 to 730 mg/l

ETC = 69.1944

$R^2 = 0.9380$

Mesoinositol is naturally present in the musts and is produced by the fermentative growth. It may be used in the calculation of the fermentability quality index  $Q_5$ .

This example indicates that the various parameters can be used with an excellent reliability to calculate the objective quality indices of the vinification compositions. The correlation coefficients are in all cases greater than 0.90; and even in most of the cases greater than 0.95, and from a relatively low

number of calibration samples. The concentration values are obtained according to the invention (from the spectrum) in less than one minute for each sample; each quality index can, for example, be calculated from the simple sum of values obtained for each characteristic parameter that makes it up, and compared to a reference value of a vinification composition considered to be of good quality 5 according to oenological theory or practice.

The invention can be the object of numerous variants in relation to the above description, which is given only by way of non-limiting example.

## PATENT CLAIMS

1. A method for the objective qualitative analysis of liquid vinification compositions, *characterised in that*:

5        • an initial configuration and calibration step (13) is carried out during which:  
          - a group of characteristic parameters are selected, the characteristic parameters  
          being capable of characterising the quality of a vinification composition to be analysed, and  
          comprising the concentration levels in the vinification composition of characteristic compounds,  
          chosen among at least a compound produced by *Botrytis cinerea*, at least a compound  
10        produced by *yeasts*, and at least a compound produced by acetic bacteria, and at least a  
          compound produced by lactic acid bacteria,  
          - calibration values ( $\alpha_i$ ,  $K_i$ ,  $B_{i0}$ ) of spectroscopic criteria are determined and recorded  
          for each characteristic parameter, the spectroscopic criteria being chosen to enable evaluation of  
          the characteristic parameter in a vinification composition from an infrared absorption spectrum,  
15        which can be generated from a sample of this vinification composition,  
          • for each vinification composition to be analysed:  
          - a spectroscopic analysis (17, 18) is carried out during which a continuous infrared  
          absorption spectrum of a sample of this vinification composition is generated,  
          - the spectroscopic criteria of each characteristic parameter are applied to the said  
20        continuous spectrum in such a way that the value ( $V_p$ ) of this characteristic parameter in the  
          vinification composition is evaluated by automatic calculation (19).

2. A method according to claim 1, *characterised in that* the group of characteristic  
parameters comprises the concentration of each of the characteristic compounds, belonging to  
25        the group consisting of gluconic acid, ethanal, ethyl acetate, arabitol, mannitol, sorbitol, 2,3-  
butanediol, methyl-3 butanol-1, glycerol, mesoinositol and isoamyl acetate.

3. A method according to any of claims 1 to 2, *characterised in that* the continuous  
spectrum is generated by Fourier transform interferometric spectroscopy.

30        4. A method according to any of claims 1 to 3, *characterised in that* the spectroscopic  
criteria are chosen to enable evaluation of each characteristic parameter in a vinification  
composition from a near and mid infrared absorption spectrum, and in that the near and mid

infrared continuous spectrum of each vinification composition to be analysed is generated.

5. A method according to any of claims 1 to 4, *characterised in* that at least an objective quality index ( $Q_j$ ) of the vinification composition is calculated as a function of the said value ( $V_p$ ) evaluated from at least a characteristic parameter of the vinification composition to be analysed.

10 6. A method according to claim 5, *characterised in* that the function is a polynomial function.

15 7. A method according to any of the claims 5 or 6, *characterised in* that there are calculated:

- a first quality index, representing the attack on the harvest by *Botrytis cinerea*, at least as a function of the value of concentration levels of gluconic acid, mannitol and sorbitol evaluated from the continuous spectrum by application of spectroscopic criteria,
- a second quality index, representing the attack by yeasts, at least as a function of the value of the concentration levels of ethanal, ethyl acetate, arabitol, of 2,3-butanediol, of methyl-3 butanol-1, of glycerol, of mesoinositol and of isoamyl acetate evaluated from the continuous spectrum by application of spectroscopic criteria,
- 20 - a third quality index representing the attack by acetic bacteria, at least as a function of values of the concentration levels of acetic acid, ethyl acetate and of 2,3-butanediol evaluated from the continuous spectrum obtained by application of spectroscopic criteria,
- a fourth quality index representing the attack by lactic acid bacteria, at least as function of the value of concentration levels of lactic acid, mannitol and of 2,3-butanediol, evaluated from the continuous spectrum obtained by application of spectroscopic criteria,
- 25 - and a fifth quality index representing the fermentation ability, at least as a function of the value of the concentration of mesoinositol.

30 8. A method according to any of claims 5 to 7, *characterised in* that each characteristic parameter is evaluated and each quality index ( $Q_j$ ) is calculated by data processing immediately after the spectroscopic analysis step has been carried out.

9. A method according to any of claims 1 to 8, *characterised in* that the spectroscopic criteria comprise for each characteristic parameter, for a selection of an Integral number N of

spectral bands  $\alpha_i$  of predetermined wavelengths, the value of the luminescence spectral density  $L_i$  of the said continuous spectrum, and in that a value  $V_p$  of the characteristic parameter is calculated according to the formula:

$$V_p = B_0 + \sum_{i=1}^N K_i L_i$$

5

where  $B_0$  and  $K_i$  are predetermined coefficients.

10. A method according to claim 9, characterised in that each  $N$  lies between 5 and 30.

10

11. A method according to any of the claims 1 to 10, *characterised in* that the group of characteristic parameters further comprises alcoholic volumetric strength; total sugar content; total acidity; pH; concentration of acetic acid; concentration of malic acid; concentration of tartaric acid; concentration of lactic acid; and content of phenolic compounds.

15

12. A device for objective qualitative analysis of liquid vinification compositions, characterised in that it comprises:

20

memory means (8) in which the calibration values  $((\alpha_i, K_i, B_0))$  of spectroscopic criteria are recorded for a group of characteristic parameters capable of characterising the quality of a vinification composition to be analysed, this group comprising the concentration levels in the vinification composition of characteristic compounds selected from at least a compound produced by *Botrytis cinerea*, at least a compound produced by yeasts, and at least a compound produced by acetic bacteria, at least a compound produced by lactic acid bacteria, the said spectroscopic criteria being selected for each characteristic parameter to enable evaluation of 25 the characteristic parameter in a vinification composition from an infrared absorption spectrum, that can be generated from a sample of this vinification composition,

30 spectroscopic analysis means (1, 2, 3, 4, 6) for collecting a sample of vinification composition and generating an infrared continuous spectrum of this sample,

30

calculation means (7) arranged to apply the spectroscopic criteria of each characteristic parameter to the said continuous spectrum and to evaluate by automatic calculation the value  $(V_p)$  of this characteristic parameter in the vinification composition.

13. A device according to claim 12, characterised in that the group of characteristic parameters comprises the concentration of each of the characteristic compounds belonging to the group consisting of gluconic acid, ethanol, ethyl acetate, arabitol, mannitol, sorbitol, 2,3-butanediol, methyl-3 butanol-1, glycerol, mesoinositol and isoamyl acetate.

14. A device according to any of claims 12 or 13, *characterised in* that the spectroscopic analysis means (1, 2, 3, 4, 6) comprise a Fourier transform interferometric spectrometer.

15. A device according to any of claims 12 to 14, characterised in that the selection criteria are chosen for each characteristic parameter to enable evaluation of the characteristic parameter in a vinification composition from a near and mid infrared spectrum, and in that the spectroscopic analysis means (1, 2, 3, 4, 6) are arranged to generate near and mid infrared continuous absorption spectra.

16. A device according to any of claims 12 to 15, characterised in that the calculation means (7) are arranged to calculate automatically at least an objective quality index ( $Q_j$ ) of the vinification composition as a function of this value ( $V_p$ ) evaluated from at least a characteristic parameter for the vinification composition to be analysed.

17. A device according to claim 16, characterised in that the function is a polynomial function.

18. A device according to any one of claims 16 or 17, characterised in that the calculation means (7) are arranged to calculate:

- a first quality index, representing the attack on the harvest by *Botrytis cinerea*, at least as a function of the value of concentration levels of gluconic acid, mannitol and sorbitol evaluated from the continuous spectrum by application of spectroscopic criteria,
- a second quality index, representing the attack by yeasts, at least as a function of the value of the concentration levels of ethanol, ethyl acetate, arabitol, of 2,3-butanediol, of methyl-3 butanol-1, of glycerol, of mesoinositol and of isoamyl acetate evaluated from the continuous spectrum by application of spectroscopic criteria,
- a third quality index representing the attack by acetic bacteria, at least as a function of values of the concentration levels of acetic acid, ethyl acetate and of 2,3-butanediol evaluated from the

continuous spectrum obtained by application of spectroscopic criteria,  
 - a fourth quality index representing the attack by lactic acid bacteria, at least as function of the  
 value of concentration levels of lactic acid, mannitol and of 2,3-butanediol, evaluated from the  
 continuous spectrum obtained by application of spectroscopic criteria,  
 5 - and a fifth quality index representing the fermentation ability, is calculated at least as a function  
 of the value of the concentration of mesoinositol.

19. A device according to any one of claims 16 to 18, *characterised in* that the calculation  
 means (7) are arranged to evaluate each characteristic parameter and to calculate each quality  
 10 index Qj by data processing immediately after the generation of the continuous spectrum by the  
 spectroscopic analysis means (1, 2, 3, 4, 6), and to provide the results of these calculations to  
 means (10) that are read by a user.

15 20. A device according to claim 19, *characterised in* that the means (10) that are read  
 comprise means for printing a report of results.

21. A device according to any of claims 12 to 20, *characterised in* that for each  
 characteristic parameter, the calibration values comprise an integral number N of spectral bands  
 of predetermined wavelengths and coefficients Ki and Bo, and in that the calculation means  
 20 (7) are arranged to calculate a value Vp of the characteristic parameter from values of the  
 luminescence spectral density Li of the said continuous spectrum obtained for the N spectral  
 bands  $\alpha_i$ , according to the formula:

$$V_p = B_o + \sum_{i=1}^N K_i L_i$$

25 22. A device according to claim 21, *characterised in* that N is a number between 5 and  
 30.

23. A device according to any one of claims 12 to 22, *characterised in* that the group of  
 characteristic parameters further comprises: alcoholic strength; total sugar content; total acidity;  
 30 pH; concentration of acetic acid; concentration of malic acid; concentration of tartaric acid;  
 concentration of lactic acid; and content of phenolic compounds.

## ABSTRACT

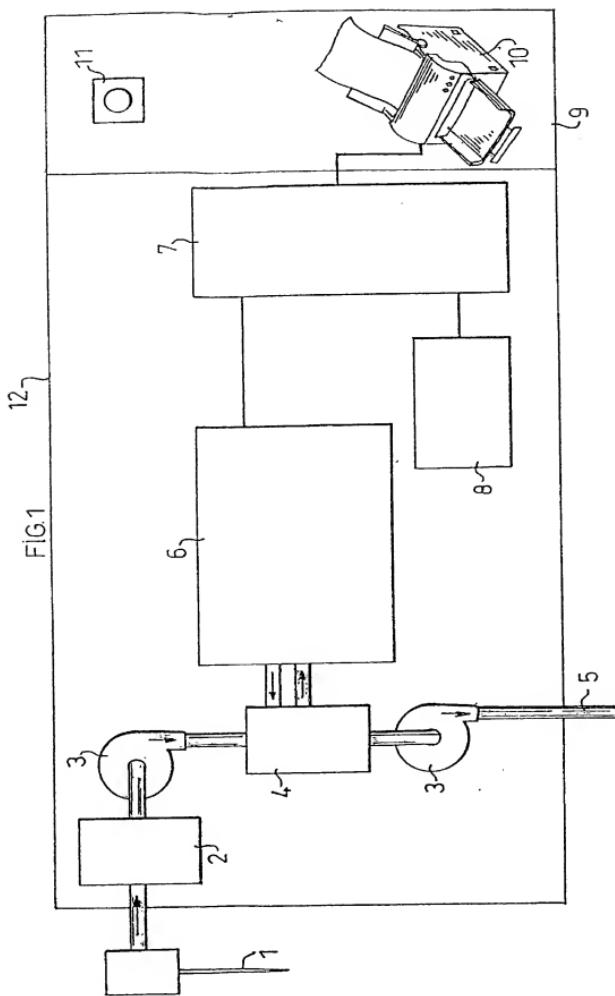
METHOD AND DEVICE FOR OBJECTIVE QUALITATIVE ANALYSIS OF GRAPE MUSTS  
AND/OR WINES BY BROADBAND INFRA-RED SPECTROMETRY

- 5 The invention concerns a method and a device for the objective qualitative analysis of liquid vinification compositions, comprising memory means (8) in which are recorded calibrating values of spectroscopic criteria for a group of characteristic parameters comprising the concentration levels of at least a compound produced by *Botrytis cinerea*, at least a compound produced by yeasts, at least a compound produced by the acetic bacteria, and at least a compound produced
- 10 by lactic acid bacteria, means (1, 2, 3, 4, 6) for producing a continuous infrared spectrum, and means (7) for calculating the value ( $V_p$ ) of each characteristic parameter in the composition from the spectrum

## ABSTRACT

### METHOD AND DEVICE FOR OBJECTIVE QUALITATIVE ANALYSIS OF GRAPE MUSTS AND/OR WINES BY BROADBAND INFRA-RED SPECTROMETRY

5 The invention concerns a method and a device for the objective qualitative analysis of liquid vinification compositions, comprising memory means (8) in which are recorded calibrating values of spectroscopic criteria for a group of characteristic parameters comprising the concentration levels of at least a compound produced by *Botrytis cinerea*, at least a compound produced by yeasts, at least a compound produced by the acetic bacteria, and at least a compound produced  
10 by lactic acid bacteria, means (1, 2, 3, 4, 6) for producing a continuous infrared spectrum, and means (7) for calculating the value ( $V_p$ ) of each characteristic parameter in the composition from the spectrum.



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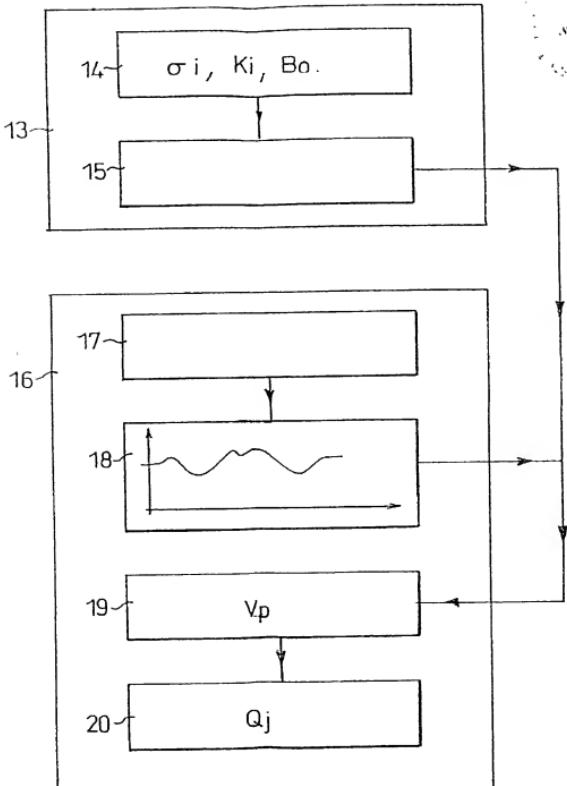


FIG. 2

## DECLARATION AND POWER OF ATTORNEY

Atty. Dkt. No.: 04590-000717

## DECLARATION

As a below named inventor, I hereby declare that:

My residence, mailing address and citizenship are as stated below next to my name,

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

**"METHOD AND DEVICE FOR OBJECTIVE QUALITATIVE ANALYSIS OF GRAPE MUSTS AND/OR WINES BY BROADBAND INFRARED SPECTROMETRY"**

the specification of which (check one)

is attached hereto.  
or  
 was filed on August 17, 2000 as Application Serial No. or PCT International Application No. PCT/DK00/00455 and was amended on \_\_\_\_\_ (if applicable).

I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to patentability as defined in 37 CFR § 1.56, including for continuation-in-part applications, material information which became available between the filing date of the prior application and the national or PCT international filing date of the continuation-in-part application.

I hereby claim foreign priority benefits under 35 U.S.C. §§ 119(a)-(d) or 365(b) of any foreign application(s) for patent or inventor's certificate, or 365(a) of any PCT international application which designated at least one country other than the United States of America, listed below and have also identified below, by checking the box, any foreign application for patent or inventor's certificate, or any PCT international application having a filing date before that of the application on which priority is claimed:

PRIOR FOREIGN APPLICATION(S)				
APPN. SERIAL NO.	COUNTRY	DATE FILED (MM/DD/YYYY)	PRIORITY CLAIM	
			Yes	No
FR 99/10627	France	08/19/1999	<input checked="" type="checkbox"/>	<input type="checkbox"/>
			<input type="checkbox"/>	<input type="checkbox"/>
			<input type="checkbox"/>	<input type="checkbox"/>

ND

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I hereby claim the benefit under 35 U.S.C. § 119(e) of any United States provisional application(s) listed below:

PRIOR PROVISIONAL APPLICATION(S)	
APPN. SERIAL NO.	DATE FILED (MM/DD/YYYY)

I hereby claim the benefit under 35 U.S.C. § 120 of any United States application(s) listed below:

PRIOR U.S. APPLICATION(S)		
APPN. SERIAL NO.	DATE FILED (MM/DD/YYYY)	STATUS - PATENTED, PENDING, ABANDONED

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under 18 U.S.C. § 1001 and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

## POWER OF ATTORNEY

I hereby appoint the following attorneys with full power of substitution and revocation, to prosecute this application and to transact all business in the Patent and Trademark Office connected therewith.

⑤  
 Thomas S. Auchterlonie Reg. No. 37,275  
 John A. Castellano Reg. No. 35,094  
 Terry L. Clark Reg. No. 32,644  
 Donald J. Daley Reg. No. 34,313  
 Gary D. Yacura Reg. No. 35,416

⑥

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## CORRESPONDENCE ADDRESS

I request the Patent and Trademark Office to direct all correspondence and telephone calls relative to this application to Harness, Dickey & Pierce, P.L.C., Customer No 30593, P.O. Box 8910, Reston, Virginia, 20195, (703) 390-3030.

1-00  
Full name of sole or first inventor: Marc Dubernet

Inventor's signature: Dubernet

Date: 15/2/2002

Residence: Chemin de Bougna, F-11100 Narbonne, FRANCE *JPK*

Citizenship: France

Mailing Address: Chemin de Bougna, F-11100 Narbonne, FRANCE